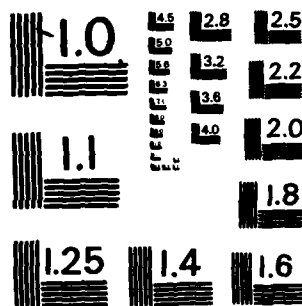


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DEPARTMENT REPORT  
UWME-DR-301-104-1

AXIAL TEST METHODS DEVELOPMENT FOR  
3-D CYLINDRICAL-WEAVE CARBON-CARBON  
COMPOSITE MATERIALS

INTERIM TECHNICAL REPORT

EDWIN M. ODOM  
DONALD F. ADAMS

AUGUST 1983

OFFICE OF NAVAL RESEARCH  
800 N. QUINCY AVENUE  
ARLINGTON, VIRGINIA  
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COMPOSITE MATERIALS RESEARCH GROUP  
MECHANICAL ENGINEERING DEPARTMENT  
UNIVERSITY OF WYOMING  
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## PREFACE

This interim report summarizes work performed on ONR Contract N00014-77-C-0503, Project No. 039-149, during the time period from September 1981 through August 1983. The Program Technical Monitor is Dr. L.H. Peebles, Jr., Office of Naval Research, Arlington, Virginia.

All work reported here was performed by members of the Composite Materials Research Group within the Department of Mechanical Engineering at the University of Wyoming. Dr. Donald F. Adams, Professor, and Mr. Edwin M. Odom are serving as Co-Principal Investigators. Students who contributed to this work include graduate students Robin L. Westberg and Robert F. Cilensek and undergraduate student Gregory V. Mehle.

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## Section 1

### Introduction

The mechanical response of carbon-carbon composite materials, and three-dimensionally reinforced carbon-carbon composites in particular, has not yet been well characterized after over a decade of active research. Progress has been hampered due to the extremely low shear strength of current carbon-carbon composites, which creates special problems in testing these composite materials. A secondary problem, but one which cannot be ignored, is that the three-dimensionally reinforced carbon-carbon composites of interest presently are fabricated utilizing a yarn orientation pattern that follows a cylindrical coordinate system. Since mechanical testing machines typically apply loadings in a rectangular coordinate system, special care must be taken to prevent nonuniform stress states in the test specimen. The main thrust of the present study has been to modify existing test methods, and to develop new approaches, for evaluating the mechanical properties of 3-D cylindrical-weave carbon-carbon composites. This work represents a continuing effort in this area [1,2].

To support this experimental work, an analytical activity has also been initiated. A full explanation of the material behavior that will be modeled analytically is included in Section 6. Additionally, this information will be used to model the various test specimens and loading configurations developed during the current effort. With the progress made in developing test methods, and the improvements in the analytical work, it is believed that a set of test methods suitable for carbon-carbon composites can now be suggested.



## Section 2

### Testing Philosophy and Materials Availability

#### 2.1 Testing Philosophy

The need for mechanical characterization of carbon-carbon composites has created difficulties for over a decade. The principle problem is that existing carbon-carbon composites have an inherent material weakness in that the shear strength between single fiber bundles is very low. Therefore, load transfer into single fiber bundles is very difficult. The one mechanical test that provides a graphic demonstration of this problem is the tension test. There are numerous examples in the literature of the failure surface of failed tension specimens being dominated by bundle pullout. The major factor in obtaining this failure is the low shear strength between the single fiber bundles.

While a valid tension test for carbon-carbon composites may be difficult to conduct, there is an increasing consensus that the Iosipescu shear test method is very accurate for measuring shear properties [1-7]. The major reason for the success of this test method in measuring shear properties of carbon-carbon composites is that it does not depend on inducing specimen loading by shear transfer mechanisms. This method induces shear loading in the specimen gage section by applying compressive loads at the specimen loading points. If instability can be controlled, compression loading in general is very dependable. It is this type of specimen loading that allows the experimentalist the possibility of measuring shear properties. It should be noted that while there is a growing consensus that the

Iosipescu shear test is a valid test for carbon-carbon composites, it or any other shear test has yet to be accepted for general use in testing composite materials by the composite materials community. The opposite of this is true for tension testing.

With this background in mind, previous approaches utilized to develop test methods were modified. The approaches previously utilized entailed the selection of various standard test methods for tension, compression, and shear testing. Numerous specimens were then prepared from the carbon-carbon billet available, and tested according to the test matrix outlined. The results of these tests were then carefully studied to determine the mechanical properties of the carbon-carbon billet, and to determine the merit of the test methods themselves. This approach was not as successful overall as it should have been. Perhaps the greatest disappointment in this approach to developing test methods for carbon-carbon composites was that often test results were highly suspect, but causes of these abnormal results could not be pinpointed with clarity. This prevented an in-depth study to alleviate problems with the test methods.

The approach utilized here to develop test methods has been to determine basic properties of the single fiber bundle. These properties were then utilized to study more complex specimens. More specifically, the approach entailed developing techniques to measure the axial stress-strain response of a single fiber bundle. After this was accomplished, more complex specimens, i.e., specimens containing many single fiber bundles, were axially tested. During this testing, continuous single fiber bundles in the direction of the applied load were instrumented with strain gages. This allowed the failure to be

monitored while in progress rather than after the specimen failed.

Shear property measurement was not included in the work. It is believed that previous work [2,6,7] has indicated that the Iosipescu shear test method provides the ability to induce a very uniform shear stress state in the gage section of the specimen, and hence very consistent results

## 2.2 Materials Availability

Two different cylindrical-weave carbon-carbon materials were tested during the present effort. The first material was obtained in the form of an approximately 60° arc segment taken from Billet Number 2208, manufactured by Fiber Materials, Inc. [1].

The second carbon-carbon material was obtained as a ring section cut from Billet Number C4X P1-2, manufactured by General Electric [1]. This billet ring consisted of four zones with different weave parameters. Processing information details for this material are presented in Table 1 of Reference [8].

### Section 3

#### Single Bundle Test Methods and Results

##### 3.1 Test Methods

The measurement of tensile properties of carbon-carbon composites has always been difficult. The difficulties arise in several areas. First, as explained in the previous section, inducing a tensile stress into the specimen is dependent on utilizing shear loading mechanisms for load transfer. Due to the low shear strengths of carbon-carbon composites, this dependence often leads to tension specimen failure modes highly dominated by shear failure rather than tensile failure. The second area of difficulty is that the emphasis at present is on carbon-carbon composites constructed in a cylindrical weave pattern. Therefore, the experimentalist is faced with trying to test a material constructed in one coordinate system with test machines and fixtures developed for a different coordinate system, i.e., the rectangular coordinate system. Under these conditions it is often difficult to determine if a test technique did yield the sought stress field in the specimen and if the specimen failed in the desired mode.

Noting the above, it was decided to perform basic tests on single fiber bundles. The results of these tests were then to be utilized to understand the results of succeeding tests on more complex specimens. To perform these single fiber bundle tests, approximately thirty single fiber bundles, approximately 140 mm (5.5 in) long were excised from the FMI billet segment cut from Billet Number 2208. The single fiber bundles were excised using a Dremel Moto Tool Model 270 hand grinder\* with a Number 409 sanding disc.\* An aluminum bracket was fabricated to

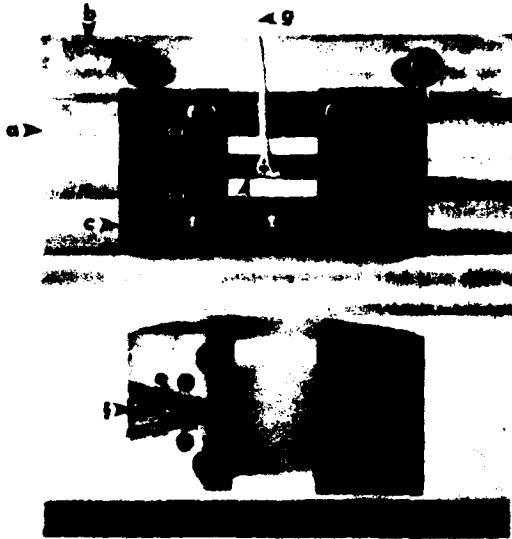
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\*Dremel, Division of Emerson Electric Company, Racine, Wisconsin.

hold the hand grinder in the chuck of a milling machine. The carbon-carbon billet was then mounted on the bed of the milling machine. The single fiber bundles were excised from the billet by moving the bed of the milling machine manually with the hand crank. Using this method, the depth of cut next to a single fiber bundle, the direction of cut, and the feed rate could be controlled without difficulty. The only problem encountered during this operation was that the carbon-carbon dust generated caused an electrical short in the hand grinder twice, necessitating repair. After a single fiber bundle was excised, its surface was manually smoothed with 400 grit emery cloth.

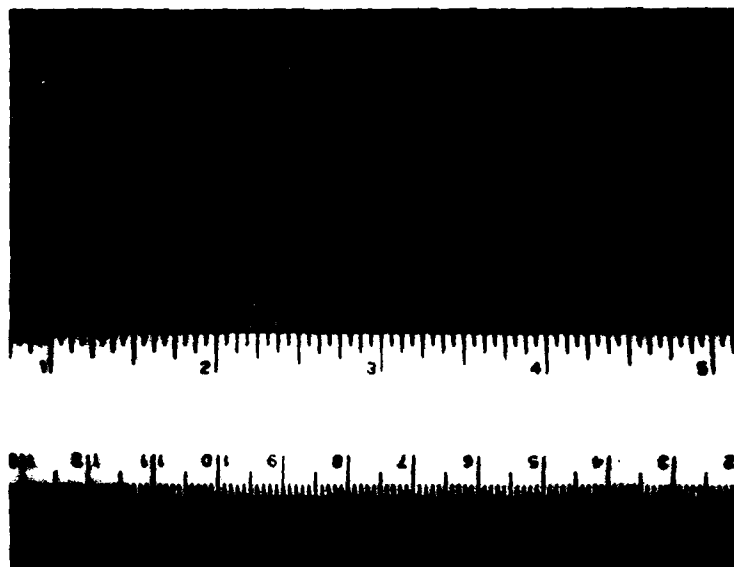
The development of the single fiber bundle tension test required several iterations. However, the specimen preparation and test fixturing initially designed performed as expected. The fixturing utilized to prepare the specimens is indicated in Figure 1. The specimen preparation procedure entailed potting the single fiber bundle into epoxy end pieces which are triangular in shape. The fixture indicated in Figure 1 was utilized to obtain specimen alignment with the test machine load frame. A finished single fiber bundle specimen is indicated in Figure 2.

After a series of specimens were prepared, tension testing was performed. This was accomplished by placing the lower portion of the molding blocks into the base plate utilized during specimen preparation (refer to Figure 1). After these blocks were properly spaced, the specimen was placed into the molding blocks. The upper portion of the molding blocks were then set in place and four Allen head cap screws were utilized to seat the two halves of the molding blocks. After checking for evidence of binding, which could damage the single fiber

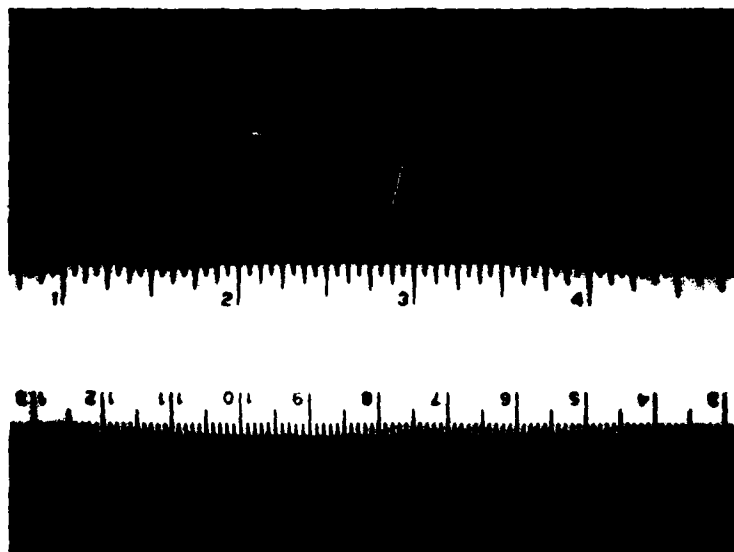


- a. Base plate of specimen preparation system. The channel is utilized to align molding blocks (c).
- b. Clamping bar utilized to hold molding blocks.
- c. Molding blocks
- d. Alignment pins.
- e. Set screw utilized to push molded epoxy ends from molding blocks.
- f. Epoxy injection post.
- g. Strain gage wire
- h. Single fiber bundle alignment jig.
- i. Adjustable set screws for centering single fiber bundle in center of molding blocks.

**Figure 1. Fixturing Utilized to Prepare a Single Fiber Bundle for Tension Testing.**



a) top view



b) side view

Figure 2. Top and Side Views of an Instrumented Single Fiber Bundle Tension Test Specimen.

bundle specimen, the specimen was tested. To test a single fiber bundle specimen, the four Allen head cap screws were removed, a steel plate was placed over the two molding blocks, and the Allen head screws were replaced. At this point the two molding blocks, the single fiber bundle tensile specimen, and the steel plate were rigidly connected together and could be removed from the specimen preparation base plate without inducing loads into the specimen. A molding block to test machine adapter was then attached to the ends of the molding blocks. This configuration is shown in Figure 3. The specimen was then ready to be inserted into the test machine load train. Prior to testing, the steel plate was removed and the Allen head screws replaced.

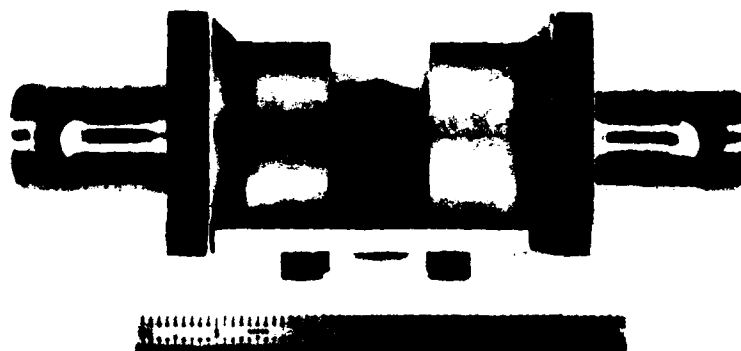


Figure 3. Single Fiber Bundle Specimen Clamped in the Molding Blocks with Test Machine Adapters in Place.



Strain data were taken during the tension testing of the single fiber bundle specimens. This was accomplished by mounting a strain gage onto the specimen. Initial attempts to mount strain gages were unsuccessful. By trial and error, however, a successful technique was developed. The principal problem was that the strain gage would not stay adhered to the specimen, or if a good bond was apparently achieved, the strain gage data were very erratic. The cause of this problem was that the surface of the specimen always was slightly rough, or had a layer of debris on it. It is important to note that this surface problem is on a micro scale, caused by the techniques utilized in surface preparation and cleaning. The strain gages utilized were Micro-Measurements EA-00-031DE-120\*. These gages are manufactured to measure strains on a material with a coefficient of thermal expansion approximately equal to zero. It was assumed that the carbon-carbon single fiber bundles would have a very low coefficient in the longitudinal direction.

To obtain good strain gage adhesion to the single fiber bundle it was necessary to specially prepare the surface before utilizing normal strain gage bonding techniques. The best method of surface preparation found was to coat the surface with M-Bond 200 adhesive. This surface preparation was performed by first cleaning the surface with Clorethane NU. M-Bond 200 catalyst was then brushed on to the surface and allowed to dry. A second coat of M-Bond 200 was then brushed on to the surface and then wiped off. Due to the chemical nature of this adhesive only a thin layer was catalyzed. However, this thin layer was sufficient to fill any surface irregularities and to immobilize surface debris on the

\*Micro-Measurements Division, Measurements Group, Raleigh, NC.

specimen surface. At this point normal strain gage mounting techniques were utilized.

### 3.2 Single Fiber Bundle Tension Test Results

All single fiber bundle tension tests were conducted on z orientation bundles excised from FMI billet Number 2208, in an Instron Model 1125 testing machine. The segment of the billet from which the single fiber bundles were excised had a length of approximately 140 mm (5.5 in) in the z-direction. After the excised specimen had been prepared for testing by the procedures outlined previously, the overall length was reduced to approximately 108 mm (4.75 in) with a gage section length of 28 mm (1.1 in).

A summary of the results of the single fiber bundle tension tests conducted are included in Table 1. These results are divided into three groups. Each of these groups of specimens was prepared differently, due to shortcomings noted during the testing sequence. The first group of specimens, i.e., Specimens 1 and 2 in Table 1, did not actually fail. The end of the single fiber bundle pulled out of the molded epoxy. After these two specimens were tested, specimen testing was suspended. Along with the problem of the specimen pulling out of the molded epoxy, the tensile modulus measured was suspected of being too high. This conclusion was based upon the fact that the FMI billet was known to have been fabricated with Hercules HM fibers. These fibers have a tensile modulus of 358 GPa (52 Msi) [8]. Using a simple rule of mixtures relation and assuming that the carbon matrix contribution to stiffness is negligible, it would be expected for an assumed 50 percent fiber volume that the resultant single fiber bundle tensile stiffness would be about 179 GPa (26 Msi) instead of the 289 GPa (41.9 Msi) average value

Table 1

## Single Fiber Bundle Tension Test Results

Specimen Number	Specimen Failure In Gage Section	Number of Pins	Calculated Strain to Failure (%)	Tensile Modulus		Ultimate Strength	
				GPa	Msi	MPa	ksi
<u>Group 1</u>							
1	No	0	20	300	43.5	603	87.5
2	No	0	24	276	40.1	675	98.0
<u>Group 2</u>							
3	Yes	1	25	308	44.7	766	111.2
4	No	1	22	334	48.5	744	108.1
5	Yes	1	26	370	53.7	968	140.5
6	No	1	26	303	44.0	787	114.3
7	Yes	1	18	400	58.0	719	104.3
8	No	1	29	277	40.2	804	116.7
Average*				359	52.1	817	118.6
Standard Deviation*				47	6.8	132	19.2
<u>Group 3</u>							
9	Yes	3	21	247	35.9	515	74.7
10	Yes	3	28	178	25.9	503	73.0
11	Yes	3	26	218	31.6	567	82.3
12	No	3	23	390	56.6	882	128.0
13	No	3	19	335	48.6	624	90.5
Average*				214	31.1	528	76.7
Standard Deviation*				35	5.0	34	5.0

\*Averages and standard deviations calculated including only specimens that failed in the gage section.

measured. This assumes that first the fiber modulus is unaffected by processing temperature and pressure, and second that a sheath of high modulus material is not formed around each fiber during the carbon-carbon processing, as has been reported in some instances [9].

Of the problems identified above, the suspected erroneous measurement of the tensile modulus was of most concern, due to past difficulties of strain data measurement. To solve this problem, all the test techniques and calibration values were verified. This effort did not identify any errors during the actual test. The only logical explanation of the higher than expected modulus was that the strain being measured was lower than the actual specimen strain at a given stress level. It was believed that this could occur if a thin outer layer of the specimen was not being strained to the same level as the remainder of the specimen. This would tend to suggest that the induced load in the specimen was not uniform through the cross section of the specimen. This implies that load transfer by a shear mechanism within the single fiber bundle is inadequate. However, if this were true it would be expected that the outer sheath of the specimen would be at a higher stress, and consequently at a higher strain level, since the load was induced only on the surfaces of the specimen by the molded epoxy and tabs. If this happened, the resultant modulus calculated from test data would be lower than the true material modulus, not higher.

To resolve the problem, several attempts were made to mount both a strain gage and an extensometer on the same single fiber bundle. It was reasoned that if the strain gage was measuring the strain of a layer of surface material that was not being strained uniformly within the remainder of the specimen, an extensometer knife edge blade would be

able to reach and measure the subsurface strains. The results of these tests were somewhat inconclusive, but did suggest that the strain gage was reading strain comparable to the extensometer. At this point, it was decided to first solve the problem of the single fiber bundle pullout and upon resolution of that problem, to return to the suspect modulus problem.

The second group of single fiber bundle specimens indicated in Table 1 were prepared differently, to try to prevent the bundle pullout problem that occurred. To prevent bundle pullout, a 0.28 mm (0.011 in) diameter hole was drilled through the bundle at each end, transverse to the length of the bundle. A 0.23 mm (0.009 in) diameter steel pin was inserted into each hole and the single fiber bundle was prepared into a specimen as outlined previously. It was hoped that the pin at each end would transfer a portion of the load into the single fiber bundle sufficient to prevent fiber pullout. The results of the attempt were mixed; of six specimens tested, three failed in the gage section and three failed in the epoxy end pieces and then pulled out. In comparing Specimens 3, 5 and 7 (gage section failures) to Specimens 4, 6 and 8 (pullout failures) in Table 1, it can be seen that the tensile modulus is higher for the specimen that had gage section failures. If it is assumed that the pin was responsible for transferring a high percentage of the load into the specimen, but that the load was not transferred efficiently across the specimen cross section, the result would be a greater magnitude of stress in the center of the specimen cross section than on the surface. This would create a situation where the modulus would be calculated to be lower than the true material modulus. However, a higher than expected modulus was measured. Additionally,

this uneven stress distribution would tend to yield specimen failure strengths lower than the true material strength. This discrepancy cannot be explained in terms of test method or shear transfer arguments.

The third group of specimens was prepared utilizing three steel pins to transfer load into the specimen on each end of the bundle. Figure 4 indicates these pins. It should be noted in this figure that the outer pins are resting on the surface in a semicircular notch. This was necessitated by the fact that if three 0.28 mm (0.011 in) diameter full holes were drilled together, they would span the total width of the single fiber bundle specimen, effectively cutting the specimen into two pieces. This pinning operation was performed in the hope of obtaining six planes, parallel to the longitudinal direction of the single bundle, across which to transfer load into the specimens. This concept is shown graphically in Figure 5. Of the five specimens tested utilizing the three-pin configuration, three failed in the gage section. The average tensile modulus of these three specimens was 214 GPa (31.1 Msi) and the average tensile strength was 528 MPa (76.7 ksi). The tensile modulus measured is considered reasonable for the materials tested; however, the strength measured for these specimens is now questionable. It would be expected that as the stress and strain field become uniform in the gage section of the specimen, that the tensile modulus would be measured more predictably and also that the strength would increase. This did not occur. The measurement with the least scatter in all these tests was the strain to failure, which typically has the most scatter.

A plot of the stress-strain curves generated for the three one-pin specimens that failed in the gage section and the three specimens utilizing three pins that failed in the gage section are indicated in



Figure 4. Single Fiber Bundle with Three Steel Pins Used to Obtain Load Transfer.

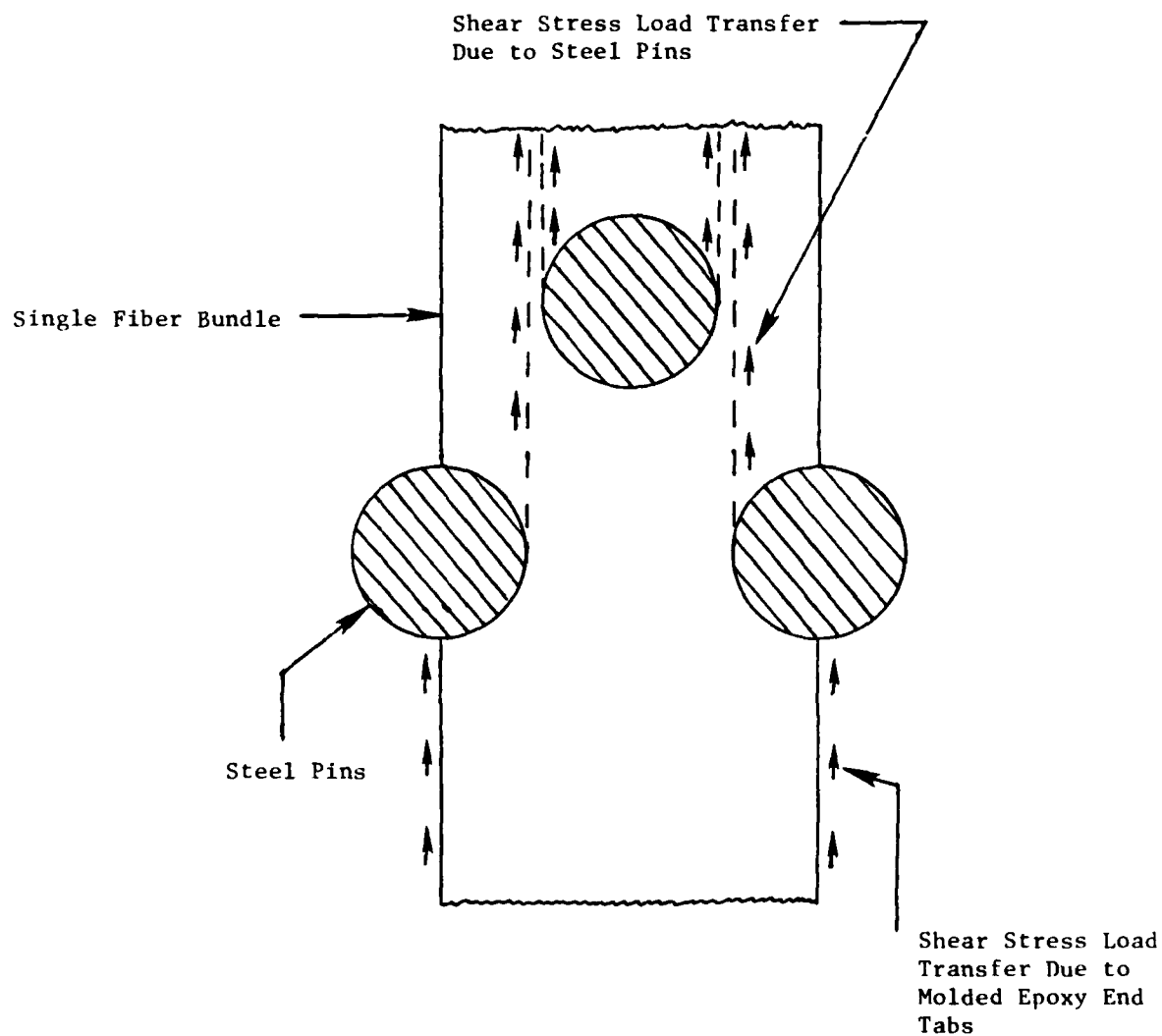


Figure 5. Schematic of a Region of a Single Fiber Bundle Indicating the Use of Three Steel Pins to Obtain Six Planes for Inducing Specimen Loading.



Figure 6. The stress-strain curves are terminated where the strain gage debonded from the specimen.

### 3.3 Conclusion

A very carefully executed attempt was made to measure the tensile strength and modulus of z-direction fiber bundles. The results overall seem very scattered. Initially, it was believed that something was not right with the test method. Subsequently, considerable attention was given to the fabrication and testing of the single fiber bundles. The results obtained, although scattered and not as would be expected, were obtained under very careful test conditions. Quite often the final explanation for other than expected test results involves the test method. This does not appear to be true in the present case.

It is strongly believed that the z-direction bundles tested in tension have varying tensile properties. There are two important aspects to this. First, the tensile modulus tended to be higher than expected. Second, there was a wide variance in the values measured. The tensile strength values measured were also other than expected. The Hercules HM fiber has a tensile strength of approximately 2.34 GPa (340 ksi). If it is assumed that the single bundle had a fiber volume of 50 percent, a reasonable bundle tensile strength value would be approximately 50 percent of the fiber strength. Specimen 5 (see Table 2) approached this value, but all other specimens failed at much lower stress levels.

As has been implied, the matrix has not been considered as adding much to the tensile properties of the single fiber bundle. Considering the results obtained, this belief may be erroneous. It has been reported [9] that a sheath of the carbon matrix material can form around

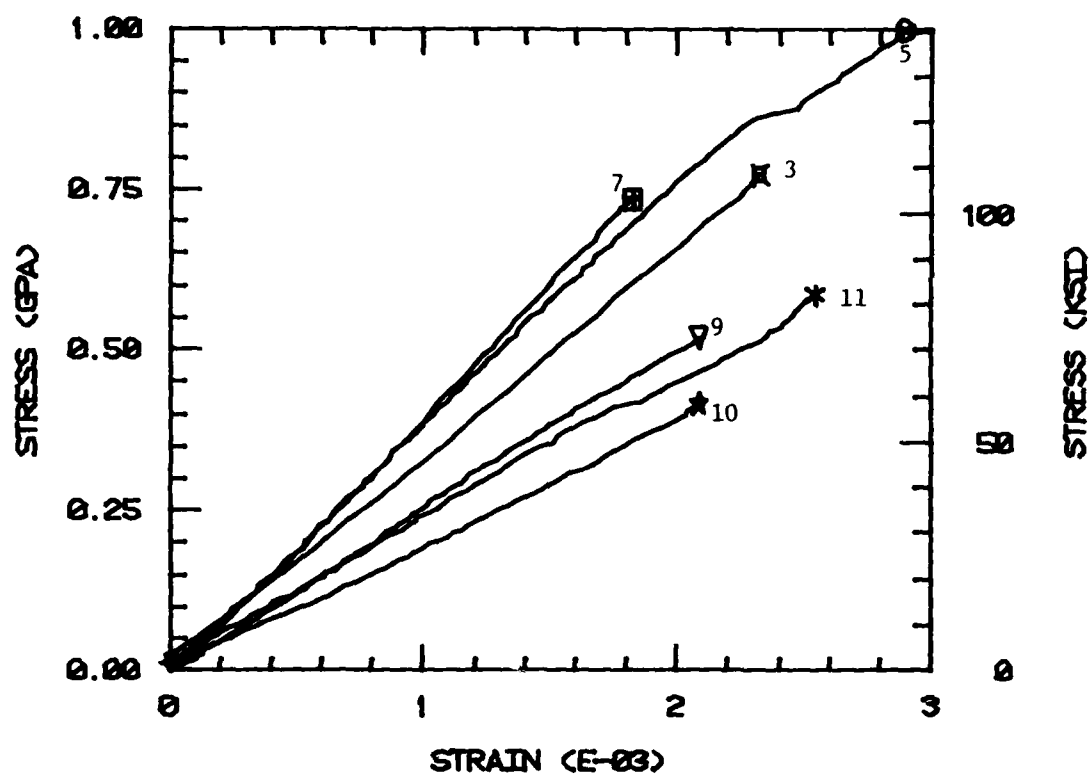


Figure 6. Tensile Stress Versus Strain Plots of All Single Fiber Bundle Tension Tests That Failed in the Gage Section (see Table 1).

each fiber, and that this sheath can have a tensile stiffness as high as 689 GPa (100 Msi). If this was the case here, the high tensile modulus would be explained. Additionally, as the modulus increases, the strength typically tends to decrease. This could explain the lower than expected strength values. If the premise of a high modulus sheath being present is accepted, its properties would be expected to be dependent on the processing temperatures and pressures. Since the single bundles tested were excised from various depths of the billet, it would be expected that these single bundles could be exposed to different temperatures and pressures during fabrication. Unfortunately, the single bundles were not identified as to their location in the billet. This should be done in future work. If the above were found to be true, it could explain the wide variance in the properties measured. The present results are not proof of the above, unfortunately.

## Section 4

### Hoop Direction Tensile Tests

In the previous work [1], two types of tension tests were performed. Complete ring tension tests were performed using fixturing similar to that described in ASTM Standard D 2290 [10]. The second tension test method utilized specimens of a different geometry. These specimens consisted of two arcs of carbon-carbon material from the same zone. The two arcs were placed back to back, with the ends potted in epoxy. A photograph of a typical specimen is shown in Figure 10. A schematic of the specimen will be presented later. Of these two test methods, the second provided more consistent results, and required much less material. Therefore, the back-to-back dual arc tension test was selected for further development.

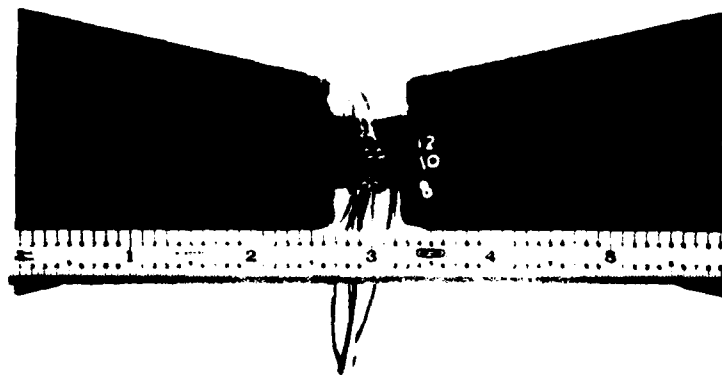
For the present study two specimens were prepared. Only two specimens were possible due to the material quality deemed necessary for a test specimen that would be utilized to study a test method. Therefore, the General Electric Billet No. C4X P1-2 was carefully studied to obtain the best site for the removal of two specimens. This site was selected to obtain symmetry of the hoop bundles in each arc, and to obtain symmetry between the two arcs. This special attention was taken to prevent self induced bending in the dual arc back-to-back specimens similar to what can occur in an unbalanced laminated composite. After the arcs were rough cut from the billet, each arc was hand-shaped to fit a template with the radius of curvature of the location of the zone in the billet. The finished arcs were then placed

back to back in a mold [1] and the ends were potted in epoxy. A finished specimen is indicated in Figure 7. Also indicated in the figure are the strain gages utilized to measure strain.

All strain gages were placed on the surface of the hoop direction bundles. It was reasoned that since the type of billet tested is to be subjected to internal pressure, the hoop direction bundles would be responsible for reacting the primary loads. Therefore, the hoop bundle behavior would be the most important to measure. Consequently, these were the bundles strain gaged. The same strain gaging technique was utilized in the dual arc back-to-back tension tests as was utilized in the single fiber bundle tension tests described previously.

The two specimens were tested in an Instron Model 1125 electro-mechanical testing machine. Both specimens were instrumented with four strain gages, to monitor the stress distribution across the gage section. However, coincident with the beginning of the test of Specimen 1, one of the gages debonded from the surface of the specimen. Therefore, this specimen had only three working strain gages. The stress versus strain plots for each specimen are indicated in Figures 8 and 9. The stress scaling was obtained by dividing the applied load by the total cross-sectional area of the hoop direction bundles, i.e., it was assumed that the radial and axial bundles did not react any of the applied load. In Figures 8 and 9 the stress versus strain curves are numbered. These numbers refer to the locations of the strain gages on the specimens, as shown in Figures 10 and 11.

Also indicated in Figures 10 and 11 are calculations of the expected failure load and the actual failure load. The expected failure load is based upon assuming all the hoop bundles are loaded in tension



**Figure 7.** Back-to-Back Dual Arc Tension Test Specimen Used To Measure Hoop Direction Tensile Properties. (These specimens are composed of two back-to-back carbon-carbon arcs, the ends being encased in molded epoxy. This specimen is instrumented with four strain gages.)

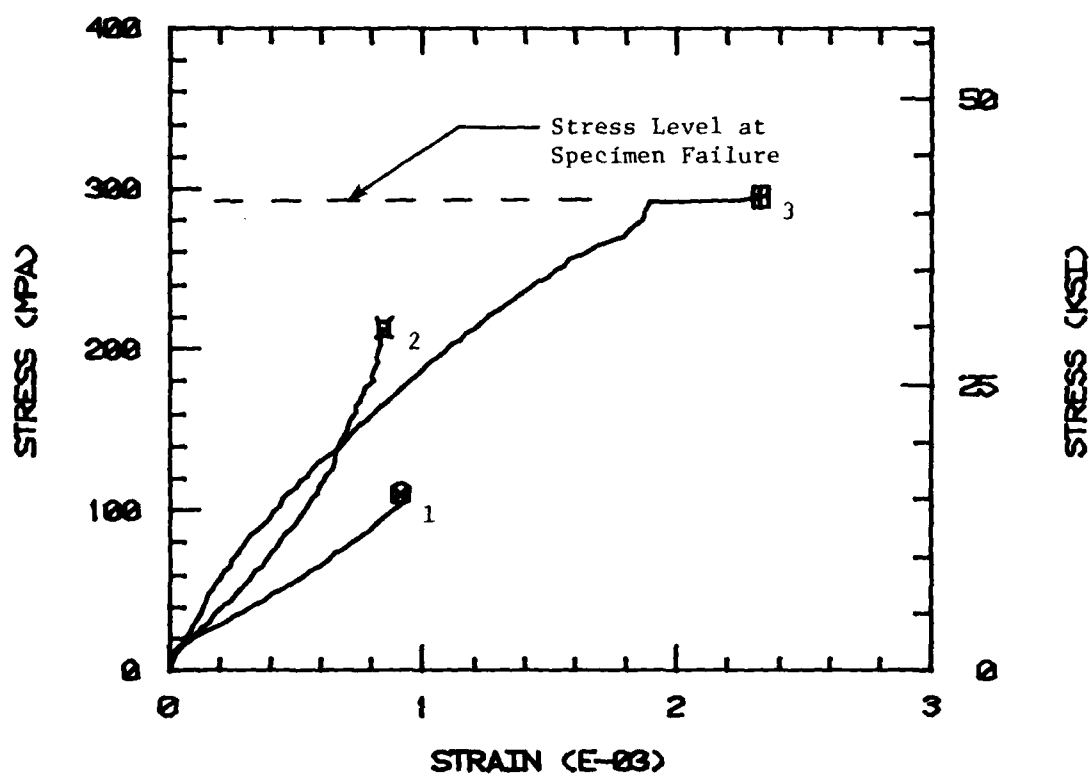


Figure 9. Stress Versus Strain Plots of Three Hoop Direction Fiber Bundles in Specimen 1 (strain gage locations are indicated in Figure 10).

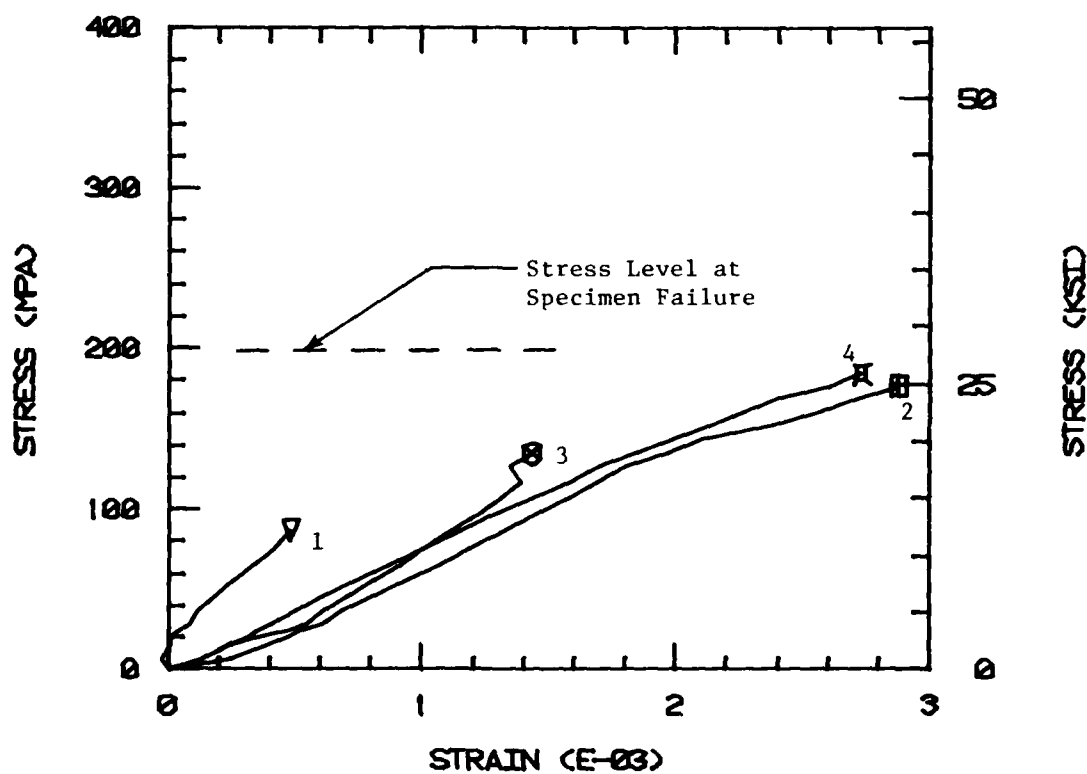
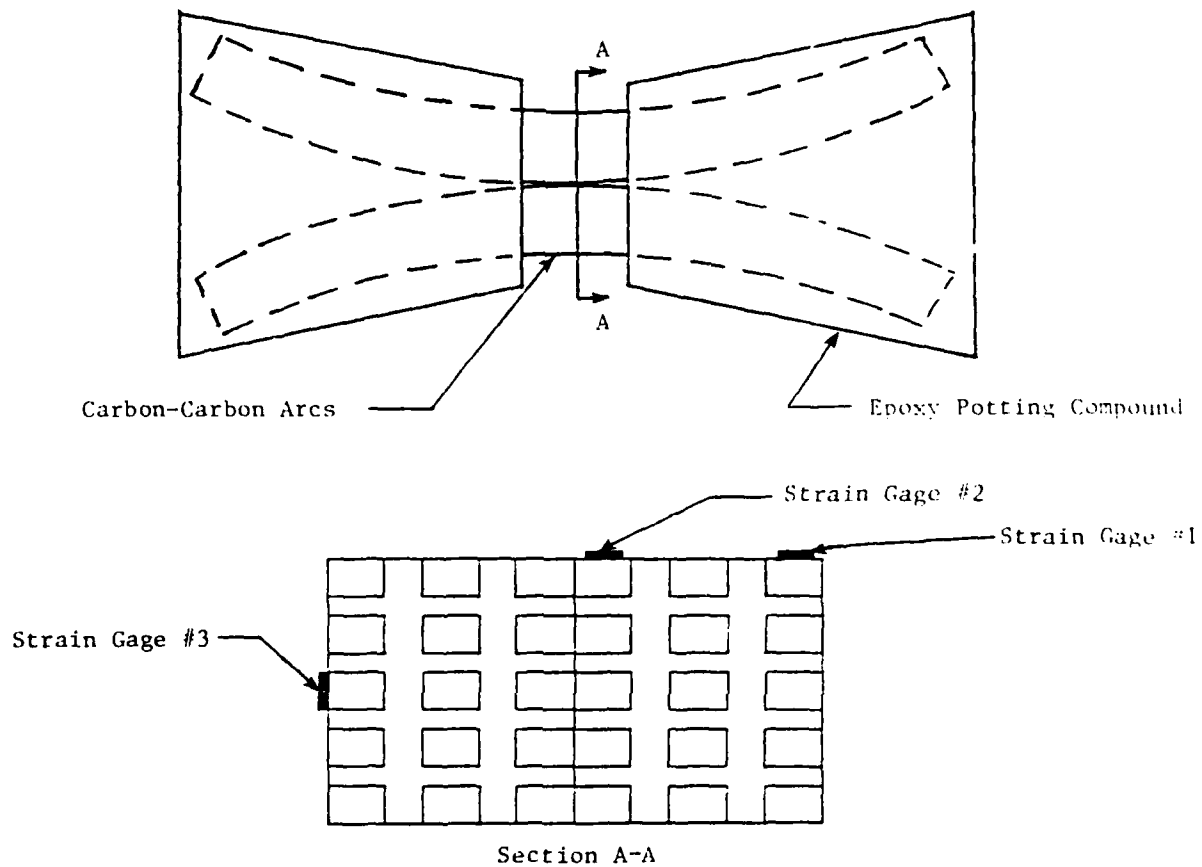


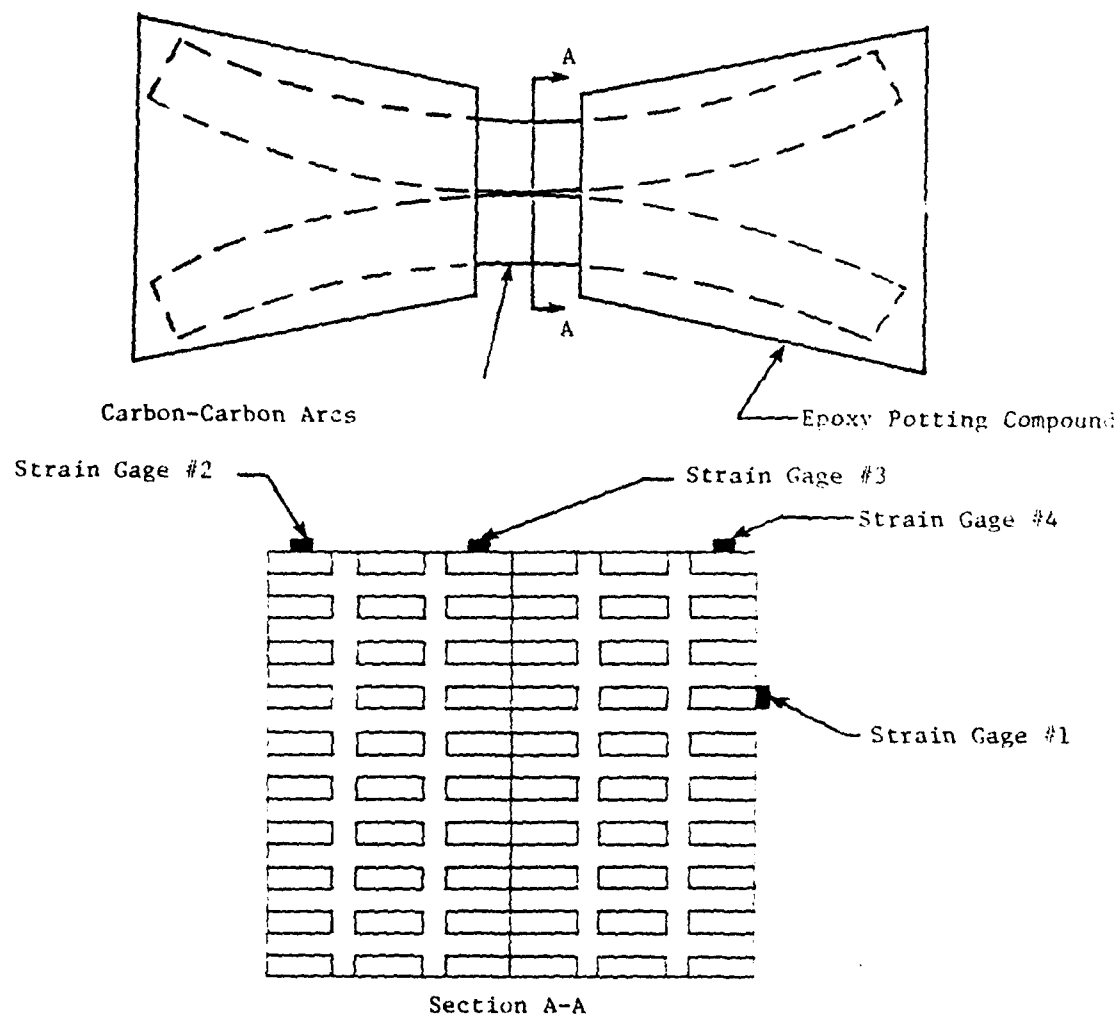
Figure 9. Stress Versus Strain Plots of Four Hoop Direction Fiber Bundles in Specimen 2 (strain gage locations are indicated in Figure 11).





Total Number of Hoop Bundles = 30  
 Average Bundle Area =  $2.19 \times 10^{-6} \text{ m}^2$  ( $0.0034 \text{ in}^2$ )  
 Bundle Strength (From Table 1) = 528 MPa (76.7 ksi)  
 For Uniform Loading: Failure Load = 34.8 KN (7823 lbs)  
 Actual Failure Load = 18.5 KN (4159 lbs)

Figure 10. Strain Gage Locations for Specimen 1.



Total Number of Bundles = 60  
 Average Area of Bundles =  $2.2 \times 10^{-6} \text{ m}^2$  ( $0.0035 \text{ in}^2$ )  
 Bundle Strength (From Table 1) = 5.28 MPa (76.7 ksi)  
 For Uniform Loading: Failure Load = 71.6 kN (16,107 lbs)  
 Actual Failure Load = 26.0 kN (5845 lbs)

Figure 11. Strain Gage Locations for Specimen 2.

evenly. In comparing Specimens 1 and 2, Specimen 1 failed at a load that was at a greater percentage of the expected load. What is believed to be the difference is the number of hoop bundles on the inside of arcs. These bundles must be loaded by a shear transfer mechanism in the carbon-carbon material. This mechanism is not reliable. If the difference between actual failure load and expected failure load is dependent on the number of subsurface bundles, then it would be reasonable to test a specimen where there are no subsurface bundles. For example, the thickness of the specimen would include two rows of hoop bundles instead of the 5 and 10 rows tested here.

Referring to Figures 8 and 9, there are two important points to note. First, the strain gages typically debond from the specimen before failure. Therefore, these stress versus strain curves are not to failure. Second, while Specimen 1 seems to have not been loaded in pure tension, Specimen 2 indicated a relatively uniform stress state. Presently, the reason for these differing behaviors is unknown. However, it is reasonable to assume that the specimen can be made to provide a uniform tensile stress state. It is believed that this specimen can be developed further, to become an accepted test method for tension testing in the hoop direction.

## Section 5

### Compression Testing

#### 5.1 Compression Test Method

Compression testing of composite materials has always presented difficulties. With carbon-carbon composites, the difficulties are even greater due to the low shear strength of the material. Standard compression tests for composites, i.e., Celanese or IITRI tests, induce specimen loading through shear mechanisms, and also tend to apply a large through-the-thickness compressive stress to the gripped section of the specimen. With the coarse structure and cracks that are inherent in carbon-carbon composites presently, these two requirements tend to rule out these standard test configurations.

During previous work [1], the Composite Materials Research Group utilized a dogboned specimen as indicated in Figure 12. The results of this testing indicated that the concept warranted further study and refinement. With the difficulties encountered when inducing a uniform shear stress in the tension specimens, as described in Sections 3 and 4, the method utilized in the previous work has the advantage of applying a compressive load to the ends of a specimen directly. Therefore, there is no dependence on inducing loads through shear loading mechanisms.

The testing of compressive specimens during the previous work did occasionally indicate an axial shear failure rather than a compressive failure. This can be attributed to end effects in the specimen. To alleviate this problem, it was decided to utilize a specimen of greater length. To prevent premature buckling of the specimen, the sides were supported. This approach was utilized previously in an unrelated study

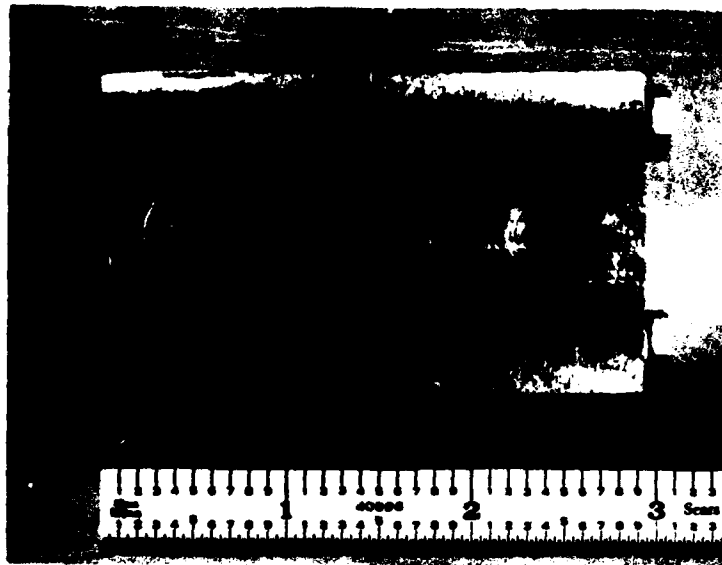


Figure 12. Compression Test Specimen Utilized During Previous Study [1].

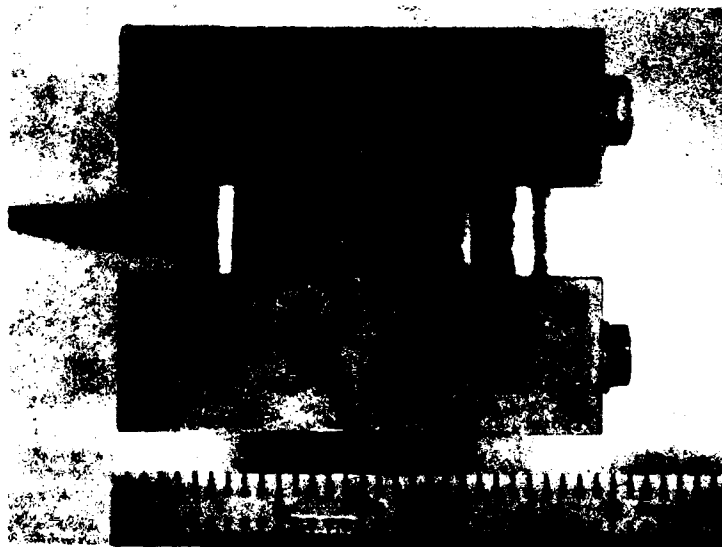
[11] with very good results.

The end loaded, side supported compression test specimen and grip configuration utilized in the present work is indicated in Figures 13 and 14. This fixture is comprised of two rectangular-shaped steel side supports. One of the supports is located at the bottom of the compression specimen, the other at the top. As is indicated in Figure 14, each of the side supports is comprised of two steel blocks bolted together. The split is needed to enable a compression specimen to be placed into the fixture and shimmed on the sides to provide a close tolerance fit. This fitting process is required since it was believed from the outset that in a test development program, the material being utilized to verify the test method must itself provide repeatable results. In terms of the test specimen itself, this implies that the specimen be machined such that the single fiber bundles in the specimen are as well aligned and symmetrical as possible. If this condition is not met, then the test degenerates from a compression test to a "bending test."

Therefore, the specimen dimensions were not dictated by the geometry of the testing fixture, but by the single bundle spacing and location. Each specimen had different geometric measurements, and had to be fitted into the specimen fixture. However, it should be noted that this fitting required shims with a thickness no greater than 0.25 mm (0.010 in). The actual fitting process is based upon judgement. A compression specimen was deemed to be fitted properly in the compression test fixture when the support fixture would no longer slide on the specimen from its own weight. It was believed that if the specimen was held any tighter than this, the portion of the specimen that is side



a) Top View



b) Side View

Figure 13. Top and Side Views of the Compression Test Fixture, With an Instrumented Test Specimen in Place.

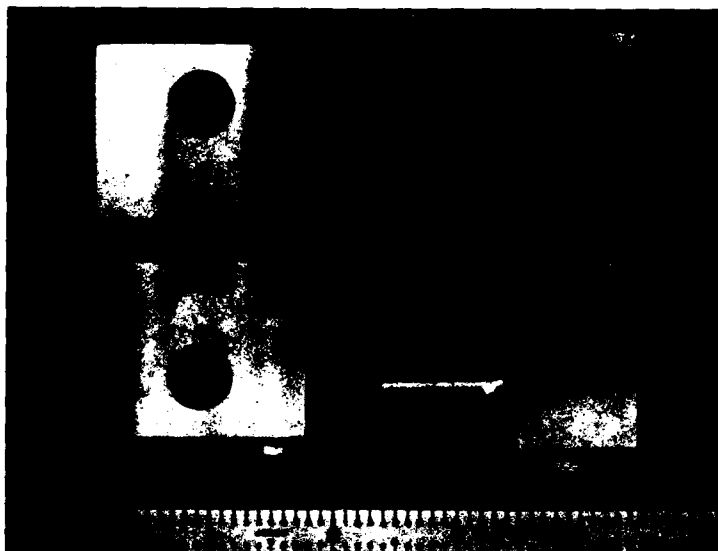


Figure 14. Compression Fixture of Figure 13 Shown Disassembled.

supported could begin to be crushed such that the test results would be compromised.

After, a specimen was fitted into the fixture, as indicated in Figure 13, the fixture was placed into an Instron Model 1125 test machine between compression platens. During the initial stages of the development of this test method, the ends of the compression fixture were covered with a 0.127 mm (0.005 in) thick layer of lead. This step was taken to provide an inspection technique, to determine if all the fiber bundles oriented in the direction of the load had been loaded during the test. It worked well for this purpose; additionally, it was found that this thin layer of lead prevented brooming of the ends of the specimen. An example of the imprint left by the end of a specimen is indicated in Figure 15. As can be seen, one row of bundles appears not



to have been loaded equally to the remainder of the bundles. This row of bundles is on the edge of the specimen, therefore an adequate lateral restraint was not available to ensure a pure compression load.

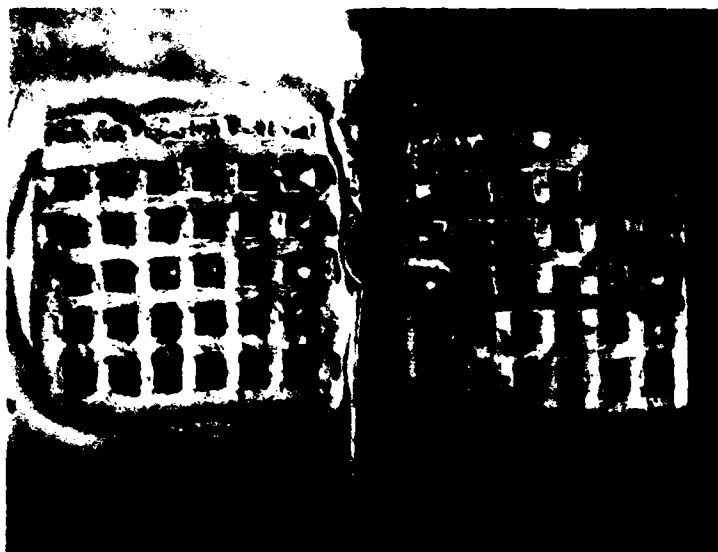


Figure 15. Thin Lead Foil (left) and the End of a Compression Specimen (right) are Shown, Illustrating the Uniformity of Specimen End Loading.

Figures 16 through 19 indicate representative examples of the compressive specimens tested. It should be noted that the specimens indicate different volume percents of bundles oriented in the axial direction. Also, the alignment of these bundles in Zones 2, 3 and 4 can be seen to change. Additionally, for the r-direction, the specimen indicates that the r-direction bundles are not continuous.

#### 5.2 Compression Test Results

Compression testing was performed in the z-direction for Zone 2, 3, and 4 materials, and in the r-direction utilizing Zone 3 material in the gage section. Testing was not performed in the  $\theta$ -direction due to the curvature of the  $\theta$  bundles. The results of this testing are presented in Tables 2 and 3. The average volume percent of the loaded bundles was calculated by taking a photograph of the specimen and then measuring the areas of the various bundles in the photograph. Table 2 indicates that as the volume percent of z-direction fibers increased for each zone, the compression strength also increased. This trend is expected since the z-direction of the specimen would have an increased axial stiffness as the volume percent of z-direction fiber bundles increased. As the stiffness increases the buckling load would be expected to increase also. However, if buckling is the major failure mechanism, which it is believed to be, the failure stress increase should have been greater than indicated by the tests. Zone 4 has only forty percent of the volume of z bundles as Zone 2, and z-direction bundle alignment would be considered poor when compared to the fiber alignment in Zone 2. Under these conditions it would be expected that the Zone 2 z-direction compression strength would be much higher. What is believed to be the reason for the small increase is that failure begins with an instability

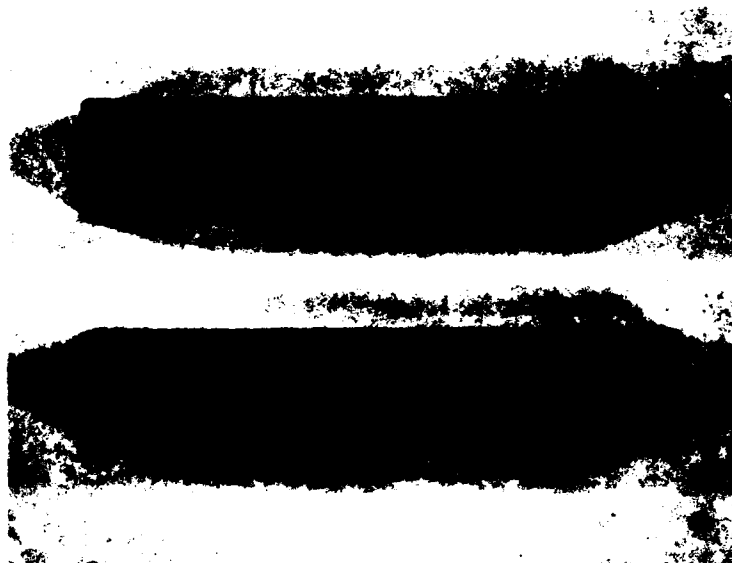


Figure 16. Representative Zone 2 Axial Direction Compression Specimens.

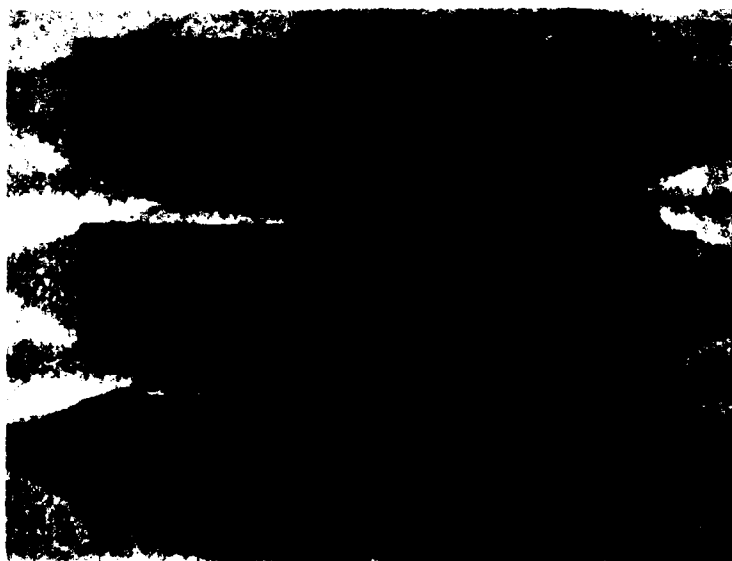


Figure 17. Representative Zone 3 Axial Direction Compression Specimens.

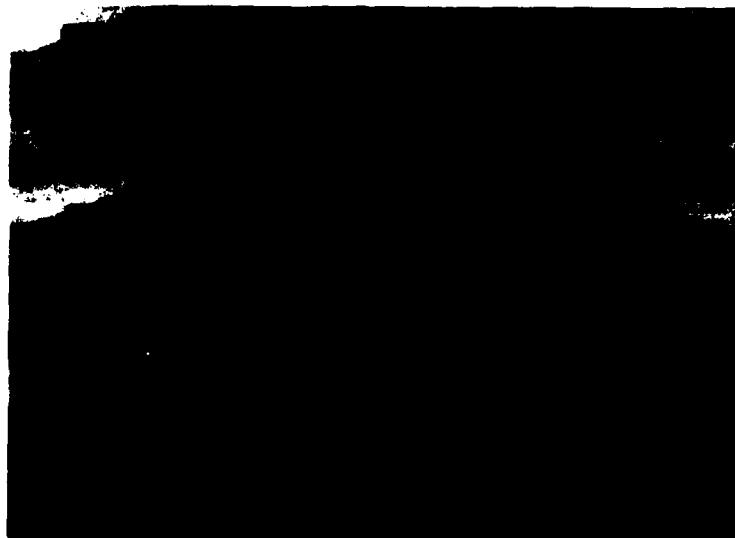


Figure 18. Representative Zone 4 Axial Direction Compression Specimens.



Figure 19. Representative R-Direction Compression Specimen.

Table 2

Specimen No.	Zone	Specimen Orientation	Average Volume		Ultimate Strength	
			Percent of	Z Bundles	MPa	ksi
1	4	z			72	10.5
2	4	z			69	10.0
3	4	z			73	10.6
4	4	z			132*	19.2*
5	4	z			76	11.0
Average				12.1	73	10.5
1	3	z			90	13.1
2	3	z			96	14.0
3	3	z			76	11.0
4	3	z			78	11.3
5	3	z			132*	19.1*
Average				17.6	85	12.3
1	2	z			60*	8.7*
2	2	z			117	17.0
3	2	z			119	17.3
4	2	z			112	16.2
5	2	z			112	16.2
Average				30.9	115	16.7

\*Not included in average

Table 3

Specimen No.	Zone	Specimen Orientation	Average Volume		Ultimate Strength	
			Percent of	R Bundles	MPa	ksi
1	3	r			67	9.7
2	3	r			68	9.9
3	3	r			61	8.9
4	3	r			71	10.3
5	3	r			70	10.1
6	3	r			56	8.1
Average				13.8	66	9.5

in the bundles that are transverse to the direction of the applied load.

The apparent trend noted above is quite interesting from a design standpoint. For example, if a high compressive strength is needed in a particular direction of the component, the solution is not to automatically increase the number of bundles in that direction. Not only does the compressive strength increase at a lower rate than the volumetric increase of bundles, but strength in the direction transverse to that direction will be decreasing. This would occur since, as the volume of bundles in one direction increases, the volume of bundles in the transverse direction has to decrease.

## Section 6

### Conclusions

Carbon-carbon composite materials are a new material that provide the designer with mechanical properties at elevated temperatures that no other material can provide. Due to this advantage, it is not a question if these materials will be utilized but a question of where and how they will be utilized. To enable these materials to be utilized, material properties must be measured and a thorough understanding of these materials must be obtained. In the past, a direct application of standard composite materials test procedures to these materials has produced in general less than acceptable results in terms of results or understanding. Therefore the approach utilized in this study was one which sought to increase the understanding or develop testing techniques and methods that would allow others to study these materials in more detail. At present, it is strongly believed that this basic approach to studying these materials should be pursued further. As the understanding is increased, the application of more standard test techniques can be implemented with modifications necessitated by the unique properties of these materials.

Emphasis during the present reporting period has been on the development of test procedures for axial properties of three-dimensionally reinforced, cylindrical weave, carbon-carbon composite materials. Three different tests were performed, i.e., single fiber bundle tension tests, hoop tension tests, and axial and radial direction compressive tests. Of the test methods developed, it is strongly believed that the compressive test method developed was the most

successful. This test should be considered for further development and utilization in determining compressive properties of carbon-carbon materials.

The tension testing performed utilizing the methods outlined needs more development. During future efforts, emphasis will be placed upon the analysis of the geometries utilized to help solve the problems encountered. This should provide the needed insight in developing a practical tension test.

Research work during the follow-on effort will be centered on doing micromechanics unit cell analyses of carbon-carbon. This will be accomplished by using known literature values for constituent material properties, as well as data measured in our own laboratories, as input to the micromechanics analysis. This three-dimensional finite element analysis will be used to predict bulk stiffness properties, damage onset, and damage progression in carbon-carbon materials.

The first phase of this follow-on work will consist of conducting an extensive literature search for mechanical properties of carbon-carbon materials and their constituents. The objective of this search will be to define thermal and mechanical properties as functions of temperature for specific types of graphite fiber bundles, as well as for the bulk graphite matrix material. It is not likely that all of the required information will be found. When possible, required unknown material properties will be measured in our laboratories. For certain material properties, e.g., transverse fiber modulus, estimates will be made. For properties which must be assumed, parametric studies will be conducted to determine the influence of that property on final material behavior.



The literature search is also expected to produce a wide variety of measured mechanical property data for carbon-carbon materials of certain specific geometries. These data will be compared with predicted results from the micromechanics analysis, to demonstrate the applicability of the analysis.

The micromechanics analysis will consist of modeling the unit cell of specific carbon-carbon weave geometries, and calculating the responses of these geometries to a variety of thermal and mechanical loadings. This analysis is a full three-dimensional finite element program developed at the University of Wyoming. The program contains capabilities for handling nonlinear, temperature-dependent material behavior, including anisotropic material response. A crack initiation and propagation capability is currently being added also. When these program modifications are completed, the analysis will be used to predict the bulk material properties, e.g., moduli and thermal expansion coefficients, for specific carbon-carbon billet designs. These results will be compared with measured values found in the literature. Damage onset by crack initiation will also be predicted. An attempt will be made to correlate these results with previous acoustic emission measurements [2] and with SEM observations [1,2,12]. Guided by the analysis, additional acoustic emission and SEM observations may be made. Crack propagation results from the analysis will be compared with cracking patterns observed by Sines, et al. [13].

The ultimate goal in perfecting such a micromechanics analysis is to produce a tool which can be used by material producers to guide constituent material and weave geometry selection. By conducting parametric studies, the importance of various geometries and processing conditions may be determined.

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Los Angeles, CA 90024

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San Antonio, CA 78200

Southern Research Institute  
Attn: Mr. H. Starrett  
2000 Ninth Avenue, South  
Birmingham, AL 35205

Southern Research Institute  
Attn: Mr. C. Pears  
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Birmingham, AL 35205

Southwest Research Institute  
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Department of the Navy  
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Nuclear Division (Y-12 Plant)  
Attn: Mr. A. Taylor  
PO Box Y  
Oak Ridge, TN 37839

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Carbon Products Division  
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PO Box 6116  
Cleveland, OH 44101

Super-Temp Company  
Attn: Mr. D. Bauer  
111205 Norwalk Boulevard  
Santa Fe Springs, CA 20360

United Technologies  
Chemical Systems Division  
Attn: Mr. R. Ellis  
PO Box 358  
Sunnyvale, CA 94088

Systems, Science and Software  
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PO Box 1620  
La Jolla, CA 92037

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Nuclear Research & Application Div.  
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Thiokol  
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Brigham City, UT 84302

Department of Mining, Metallurgical  
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University of Washington  
Seattle, WA 98195

Project Manager  
Trident Systems Project (CNM-PM 2)  
Attn: Mr. J. Crone (Code HM-2-001)  
Department of the Navy  
Washington, DC 20360

Mr. R. Wilson  
Code R31  
NSWC/WOL  
Silver Spring, MD 20910

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